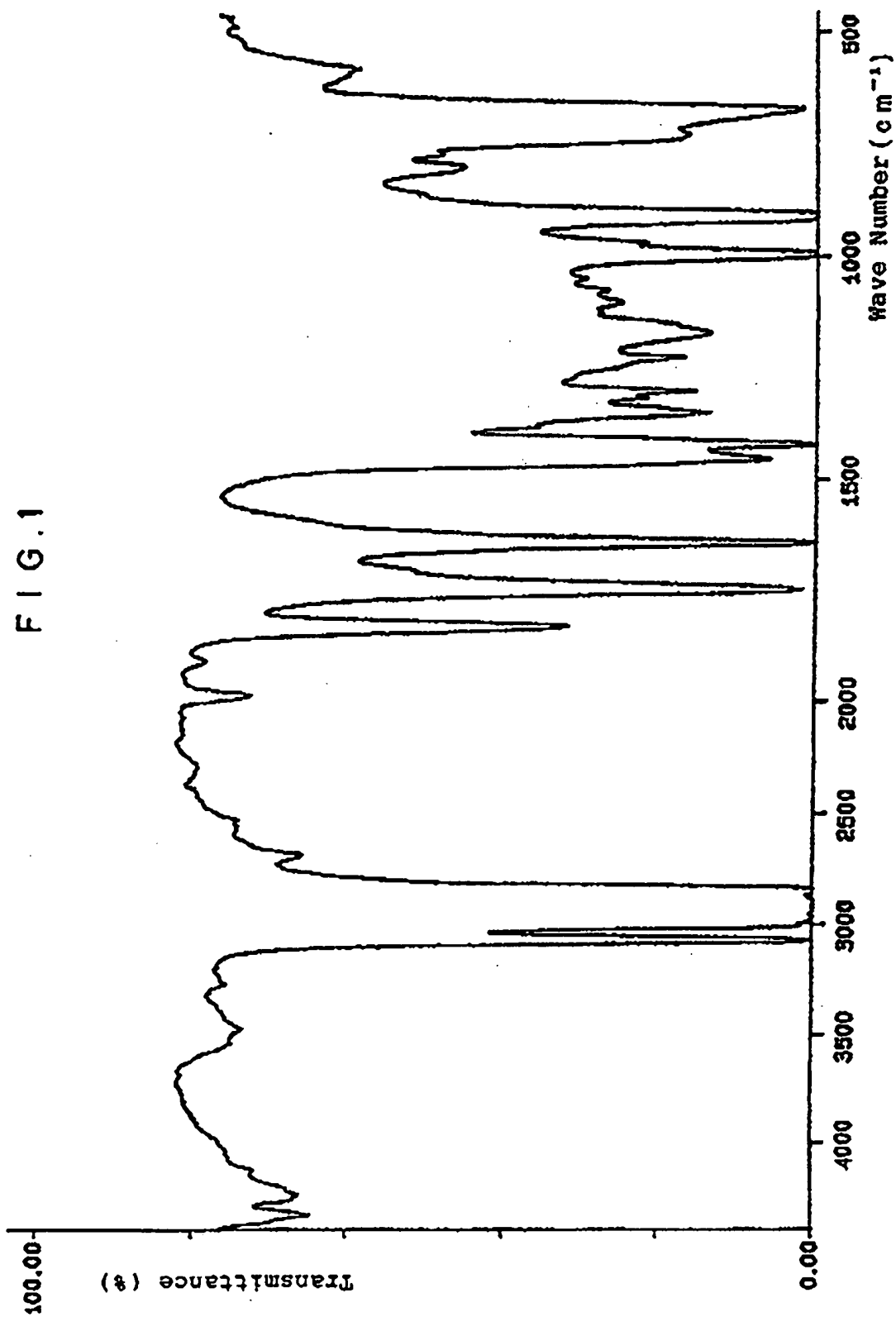


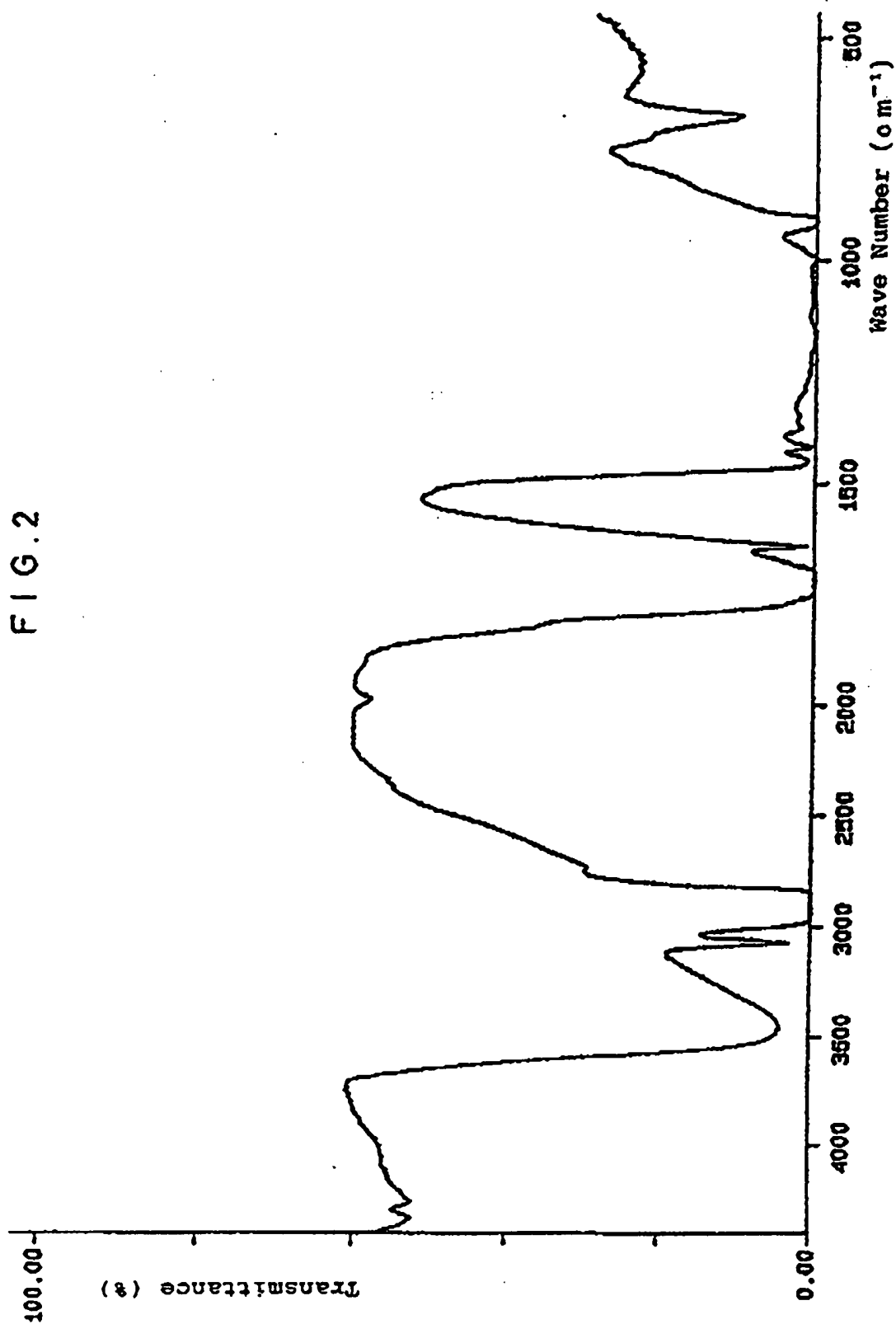
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M167 M202****U1S S1219 S1220 S1574 S1699 S1700 S3025 S3055****(56) Documents Cited****GB 2205104 A GB 1518324 A GB 0711470 A****US 4746695 A****WPI Abstract Accession No.89-124675/17 &****HU T47622A****WPI Abstract Accession No. 83-770227/38 &****SU975761 A****(58) Field of Search****UK CL (Edition N) C3M MFC MXC****INT CL⁶ C08K, C08L****ONLINE WPI****(54) Self-crosslinking composition, a product obtained from the composition, and method for manufacturing the product****(57) A self-crosslinking composition comprises: (a) an oil containing an unsaturated fatty acid and/or its ester, (b) a drying agent, (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide and optionally (d) a filler. Products such as floor tiles, elongated floor covering materials, wall covering materials, water-proof sheets may be formed from the composition.****GB 2 289 682 A**

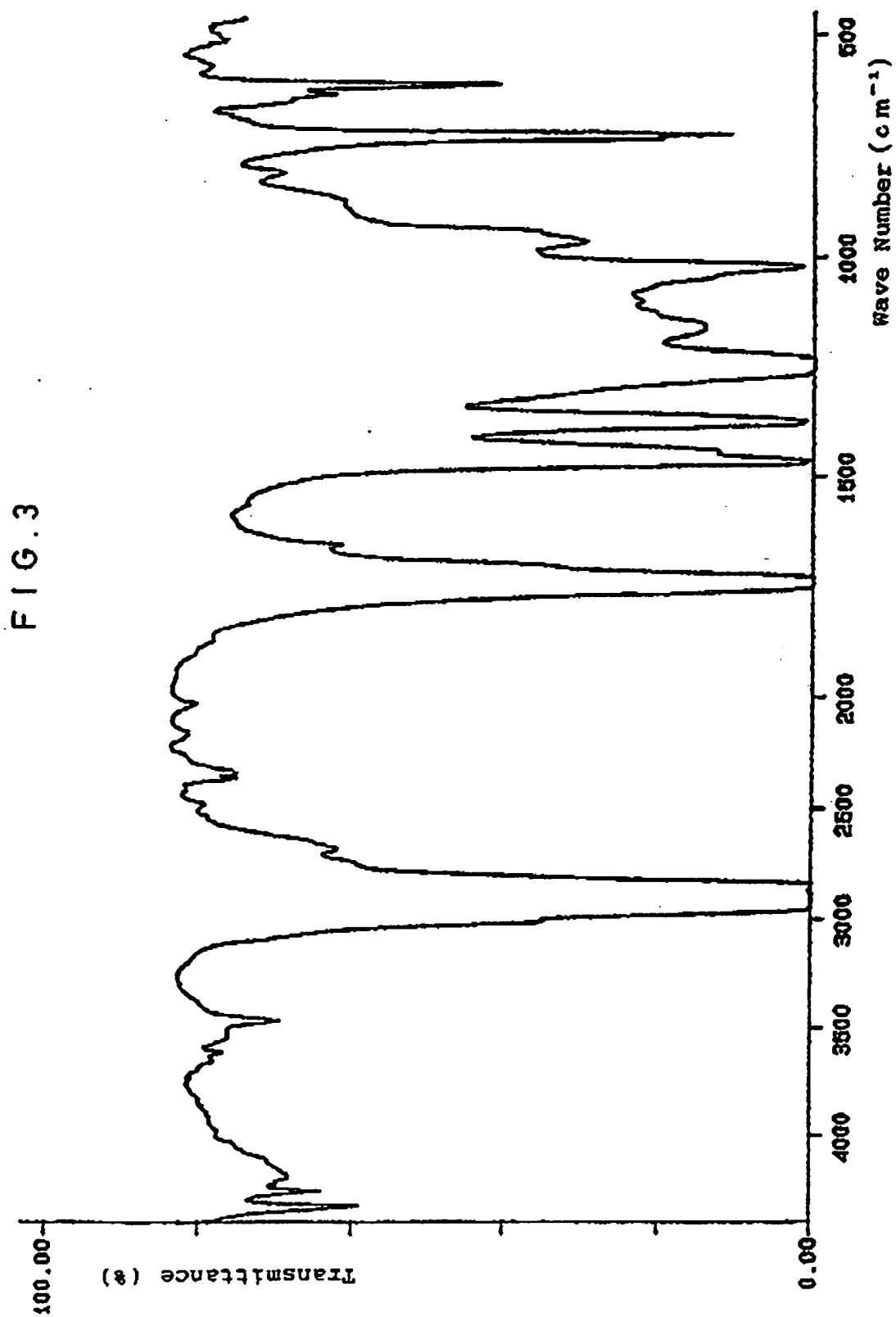
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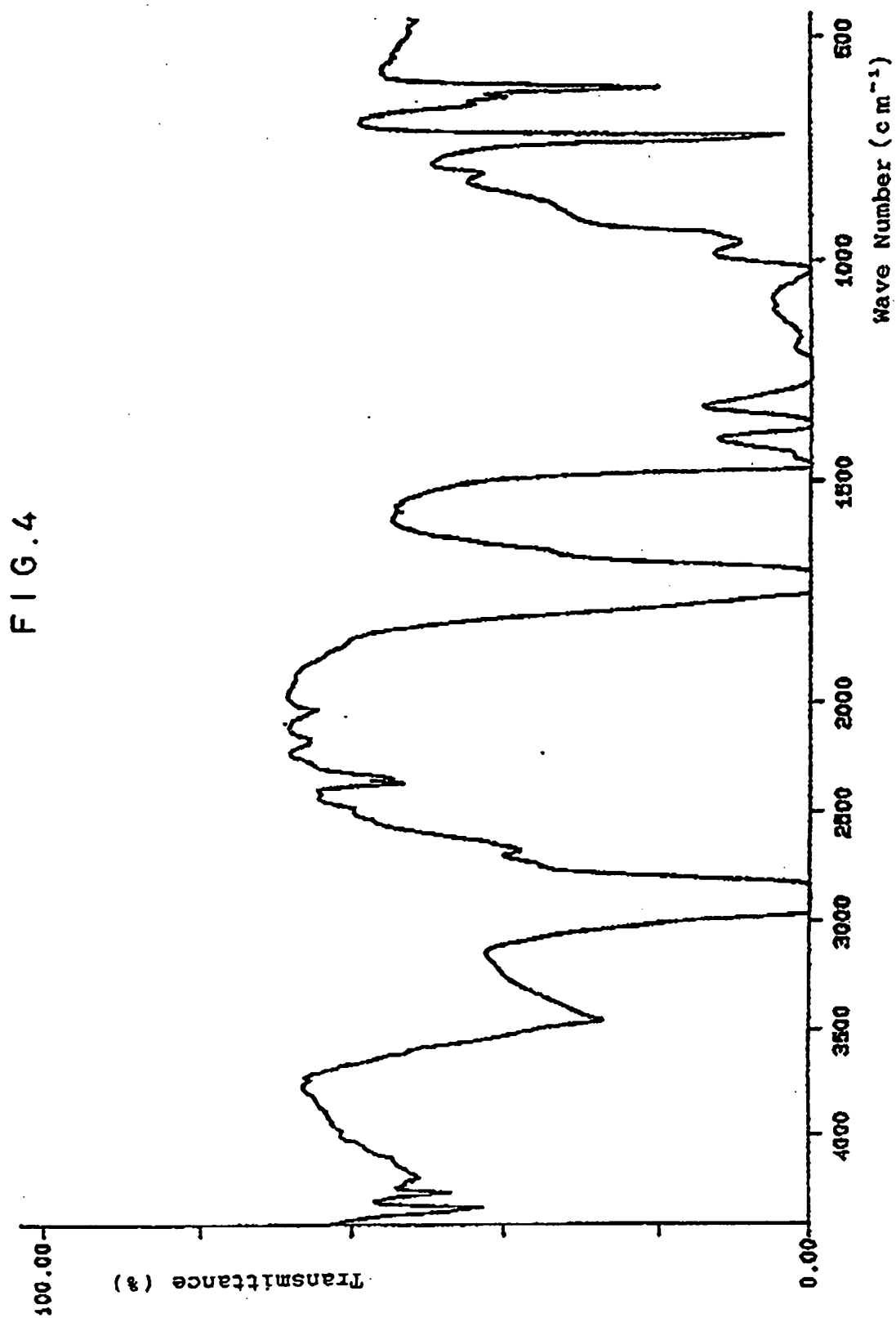
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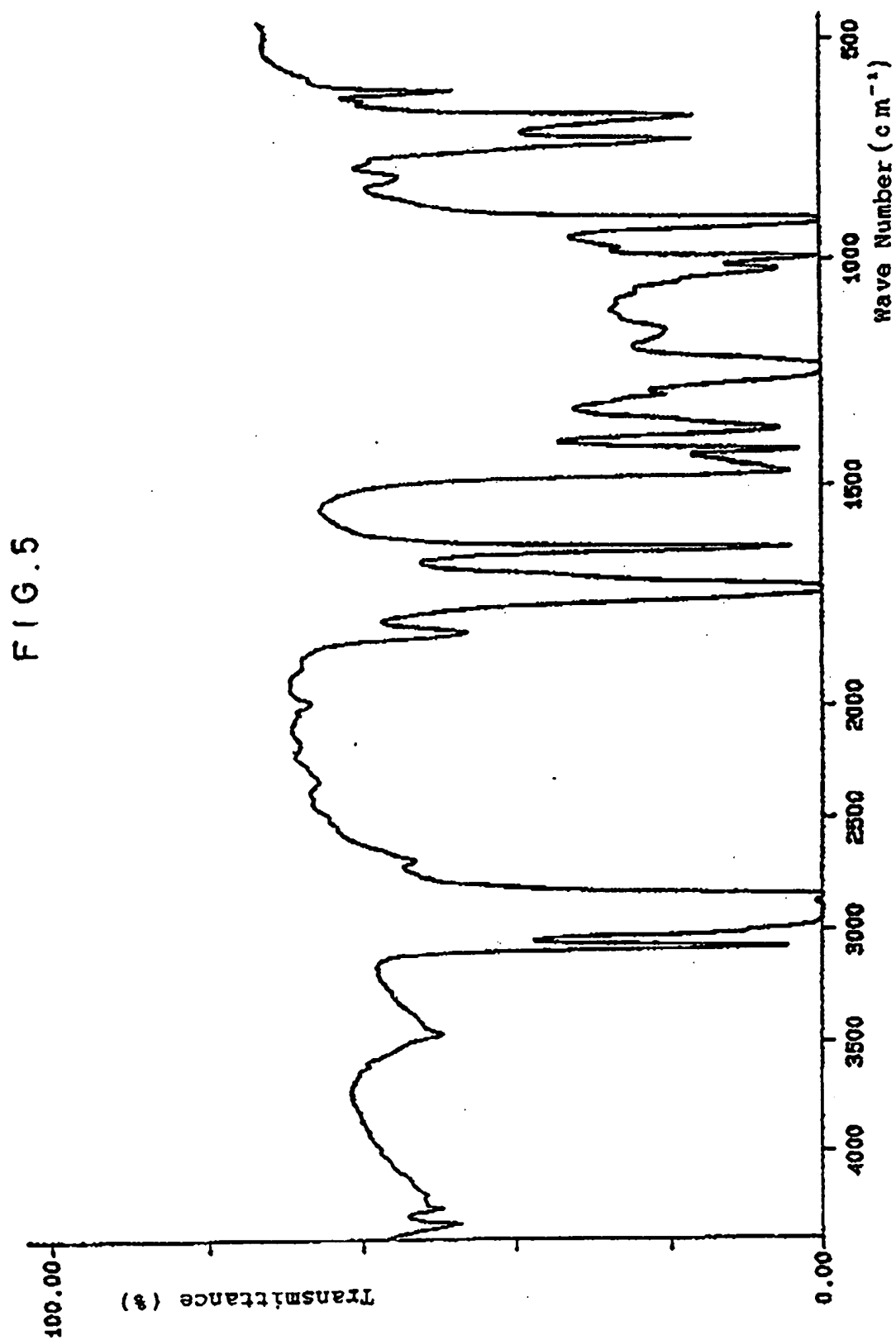
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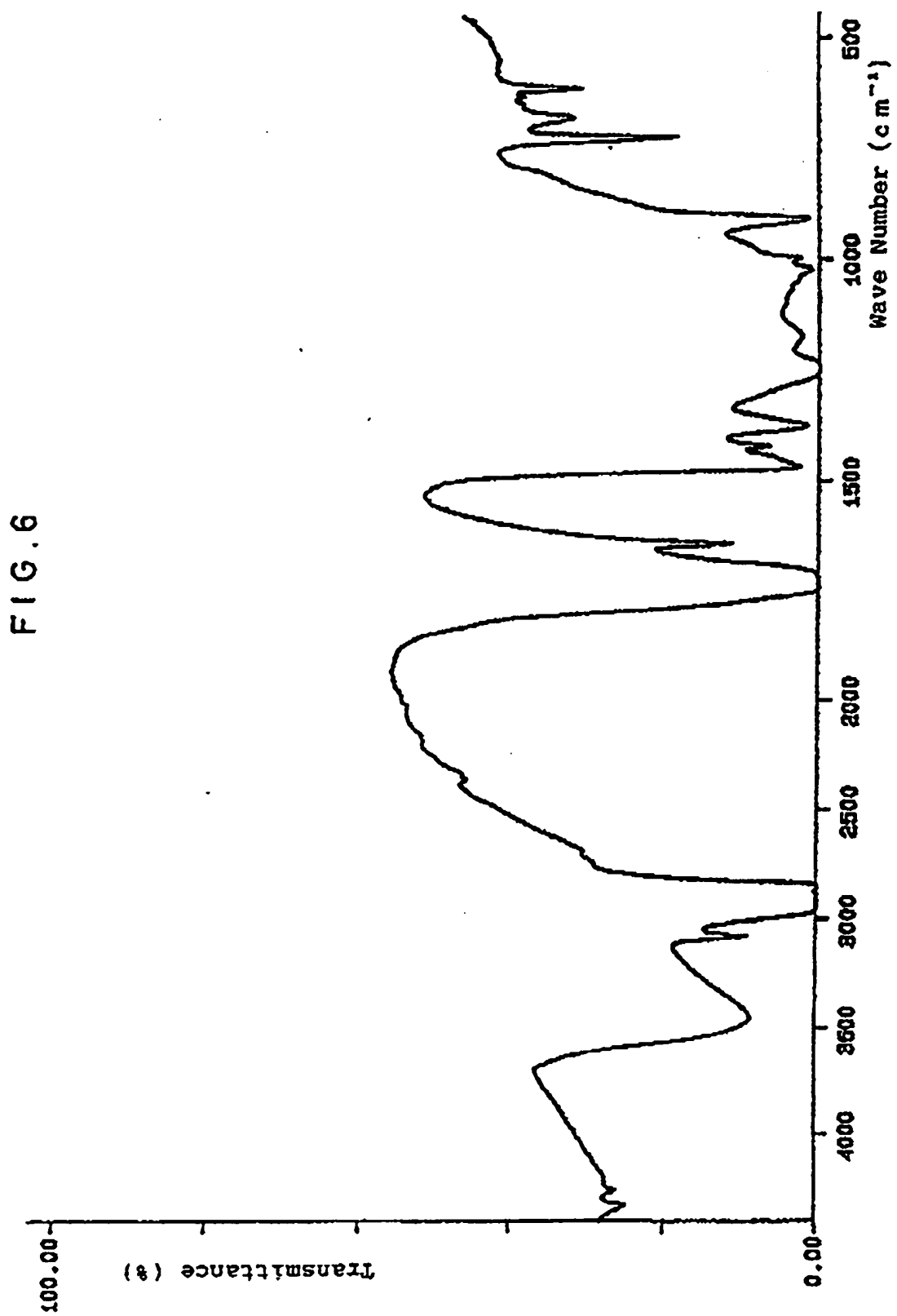
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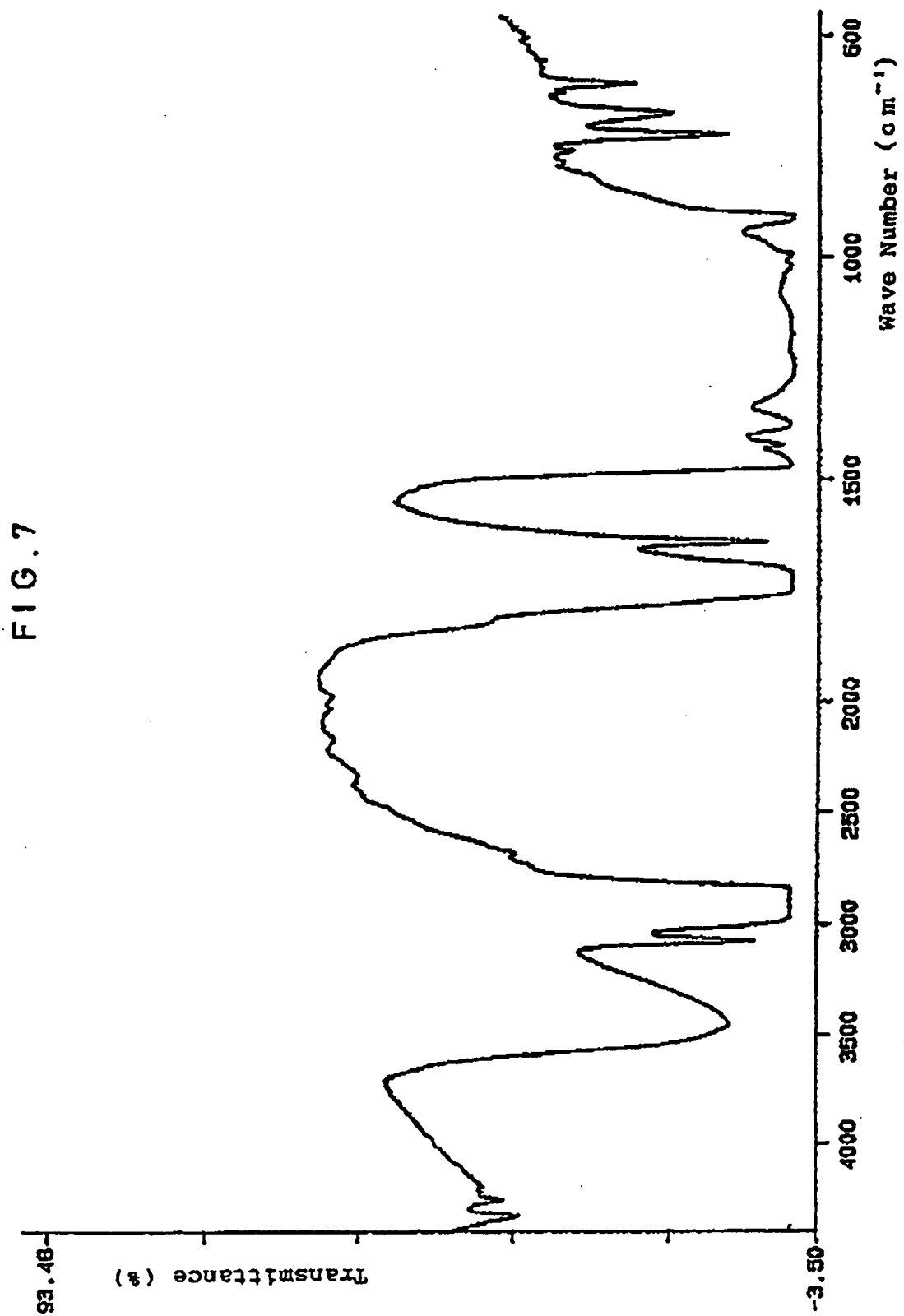
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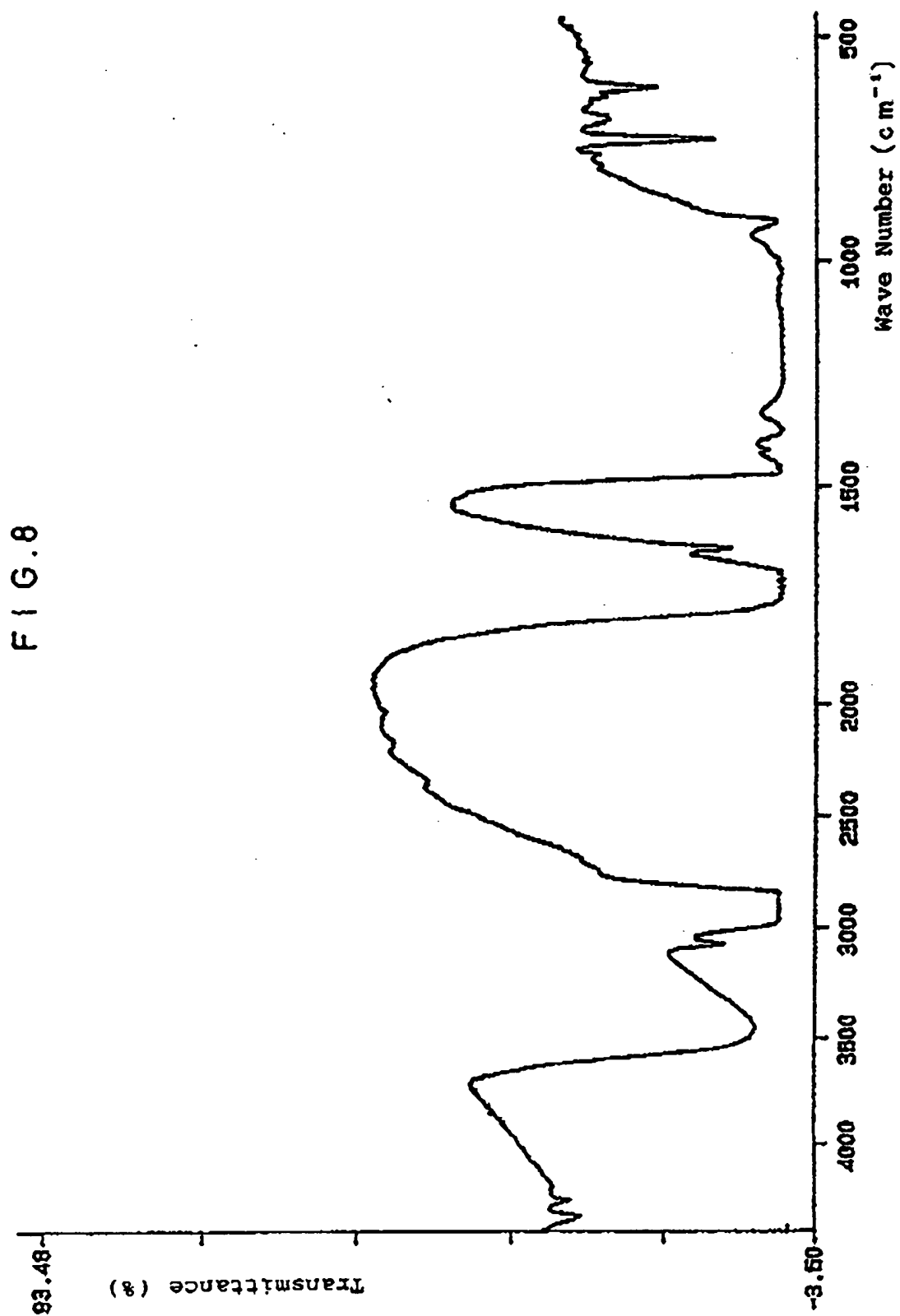
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**SELF-CROSSLINKING COMPOSITION, A PRODUCT OBTAINED
FROM THE COMPOSITION, AND METHOD FOR
MANUFACTURING THE PRODUCT**

This invention relates to a self-crosslinking composition, a product obtained from the composition, and a method for manufacturing the product. More specifically, this invention relates to a self-crosslinking composition which crosslinks a polymer or elastomer without the use of peroxide.

It is known to crosslink a polymer or elastomer using a peroxide. Such a known method, which is a crosslinking method using no sulfur, is employed to crosslink various types of elastomers and polymers except for certain polymers such as polyisobutylene. Peroxide crosslinking has the advantages of:

1. less scorch occurrence
2. shorter crosslinking time
3. superior in heat resistance and ageing resistance
4. less permanent compressive strain, and
5. free from metal contamination caused by sulfur.

There are, however, the disadvantages of:

1. the need to exclude air in order to prevent oxygen from inhibiting the generation of radicals which are required for crosslinking,

2. the inability to use process oil and anti-ageing agents because they inhibit the crosslinking action of peroxide, which makes the selection of additives difficult,

3. the need to exercise a strict control of process conditions for conducting crosslinking while excluding air. For example, if a Banbury mixer is used for kneading, the decomposition of the added peroxide caused by generated heat and the accompanying crosslinking must be prevented. Also when the materials are fed to an extruder, a careful control of the extrusion conditions is required to prevent premature solidification of polymer at the die opening, which causes serious problems.

The object of this invention is to provide a novel self-crosslinking composition which has the above-described advantages of peroxide crosslinking but which obviates or mitigates the above disadvantages of peroxide.

The first invention of this application is to provide a self-crosslinking composition which comprises:

- (a) an oil containing an unsaturated fatty acid and/or its ester,
- (b) a drying agent, and
- (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide.

The second invention of this application is to provide a product obtained by shaping and crosslinking a self-crosslinking composition which comprises:

- (a) an oil containing an unsaturated fatty acid and/or its ester,

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- (b) a drying agent,
- (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
- (d) a filler.

The third invention of this application is to provide a method for manufacturing a product by:

blending

- (a) an oil containing an unsaturated fatty acid and/or its ester,
- (b) a drying agent,
- (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
- (d) a filler.

to prepare a self-crosslinking composition;

forming the self-crosslinking composition into the required shape at a temperature lower than the crosslinking temperature of the composition; and

crosslinking the shape at a temperature higher than the crosslinking temperature of the composition.

Examples of unsaturated fatty acid used in the present invention are:

oleic acid series, $C_nH_{2n-2}O_2$, such as tiglic acid, hypogaeic acid, oleic acid, lipidic acid, petroselinic acid, erucic acid; linoleic acid series, $C_nH_{2n-4}O_2$, such as linoleic acid, tariric acid; linolenic acid series, $C_nH_{2n-6}O_2$, such as linolenic acid, eleostearic acid, tricosane; $C_nH_{2n-8}O_2$ series, such as isanic acid and arachidonic acid.

Esters of the above-listed unsaturated fatty acids and alcohols such as methanol, ethanol, propanol, butanol, and glycol, and glycerin may also be used.

Examples of oils containing the above-described unsaturated fatty acid and/or its ester are: dry vegetable oil such as hempseed oil, linseed oil, perilla oil, tung oil, walnut oil, poppy seed oil, soya bean oil, and sunflower oil; semi-dry vegetable oil such as mustard oil, sesame oil, corn oil, rapeseed oil, rice oil, and cotton seed oil; processed oil of above-listed oils; and food waste oil.

The drying agent used in this invention means one which enhances the oxidation of the above-described oil in air. Examples of drying agents are: metallic salts of naphthenic acid, resin acid and fatty acids, including salts of Co, Ni, Fe, Mn, Zn, Zr, and Pb; oxides of these metals; hydroxides of these metals; or other inorganic salts.

Examples of the polymer and/or elastomer capable of being crosslinked by peroxide include styrene-butadiene rubber (SBR), styrene-butadiene-acrylonitrile copolymer (NBR), styrene-butadiene-styrene block copolymer (SBS), styrene-isoprene-styrene block copolymer (SIS), styrene-ethylene-butylene block copolymer (SEBS), ethylene-propylene-diene terpolymer (EPR), ethylene-vinyl acetate copolymer (EVA), ethylene-ethyl acrylate copolymer, low density polyethylene (LDPE), very low density polyethylene (VLDPE), polybutadiene, and 1,2-syndiotactic polybutadiene.

If the above-described polymer or elastomer does not have a double bond within the molecule, or if it has a low crosslinking efficiency, the

main chain may include various types of diene components such as butadiene and divinylbenzene to form 1,2-syndiotactic polybutadiene, for example, and may include a monomer or oligomer having at least one functional group, such as acrylic acid, methacrylic acid, or esters thereof.

The above-described polymer and/or elastomer may be used singly or in combination as a mixture. In the latter case, a polymer of low crosslinking efficiency such as ethylene-vinyl acetate copolymer may be used with one or more polymers of high crosslinking efficiency such as 1,2-syndiotactic polybutadiene, or monomers or oligomers containing functional groups such as acrylate or methacrylate.

The type of filler used in this invention may be selected depending on the type of required product and on the type of polymer and/or elastomer. The filler may be an inorganic powder such as calcium carbonate powder, clay powder, silica powder or mica powder, or it may be organic powder such as wood powder or cork powder, or may be an organic or inorganic fibrous material.

Furthermore, the self-crosslinking composition of this invention may contain other additives than the above-described ones to an extent within the scope of the object of this invention. For example, pigment, lubricant, reinforcing agent, anti-ageing agent, fire retardant, anti-static agent may be added.

The preferred amount of oil used in this invention is in the range of from 5 to 20 weight parts (hereinafter referred to simply as "wt. parts") per 100 wt. parts of the crosslinkable component (polymer and/or elastomer).

The preferred amount of drying agent used in this invention is in the

range of from 0.005 to 1.0 wt. part per 100 wt. parts of the oil.

Although the amount of filler used to manufacture the product may be widely varied depending on the type of product, it is normally used in a range of from 20 to 500 wt. parts per 100 wt. parts of the crosslinkable component.

The crosslinking efficiency E in the presence of the crosslinkable component, organic peroxide, and crosslinking agent is expressed by the following equation,

$$E = V/2 [RO\cdot]$$

where V = crosslink concentration (mol/cm^3) in the crosslinked polymer

$RO\cdot$ = concentration of organic peroxide radicals
(mol/cm^3).

The crosslinking efficiency E becomes 1 when 1 mol of peroxide generates 1 mol of crosslink. The relation between crosslinkable component and crosslinking efficiency E is given in Table 1.

Table 1

Crosslinkable component	Crosslinking efficiency E
SBR	12.5
Cis-polybutadiene	10.5
NR (natural rubber)	1.0
NBR	1.0
CR (neoprene)	0.5
PE	1.0
EVA (ethylene-vinyl acetate copolymer)	1.0
EPDM	1.0 - 2.5
1,2-polybutadiene	90 - 100

Saturated fatty acids and esters thereof are resistant to oxidation at room temperature as compared with unsaturated compounds. Unsaturated compounds are subject to auto-oxidation following the scheme given below to generate hydroperoxide. The auto-oxidation proceeds at room temperature with a low activation energy.

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(Scheme)



Oxidation point within molecule



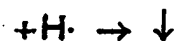
Generation of free radical



(. denotes free radical)



Peroxy-free radical



Hydroperoxide

The drying agent used in this invention is believed to generate radicals RO· rapidly from hydroperoxide and further enhance the radical-forming reaction.

Radicals thus generated induce the crosslinking reaction of the mixed polymer or elastomer similar to the crosslinking reaction performed by ordinary peroxide. The phenomenon has been confirmed by infrared spectroscopy in which a continuous reduction in the number of double bonds and an increase in the gel formation rate of the self-crosslinking composition of this invention in a hot air atmosphere have been observed.

As described above, the manufacture of the product according to this invention is conducted by mixing, forming, and crosslinking (a) an oil containing an unsaturated fatty acid and/or its ester, (b) a drying agent, (c) a polymer and/or an elastomer as the crosslinkable component, (d) a

filler, and, if necessary, other additive such as pigment and lubricant. In the processing stage, the above-described components may be added in sequence or all at one time, or may be added arbitrarily with two or more of them followed by mixing them with other component(s). When (a) the oil and (b) the drying agent are mixed together in advance and then the mixture is mixed with other components, the crosslinking reaction proceeds under a favourable condition. When a mixture of (a) the oil and (c) the polymer is mixed with a mixture of (b) the drying agent and (d) the filler, a more uniform mixture is easily prepared.

For example, the manufacture of the product of this invention is carried out by: preliminary mixing (a) the oil containing unsaturated fatty acid and/or its ester, (b) the drying agent, and (d) the filler in a Henschel mixer; mixing the prepared mixture with (c) the polymer and/or elastomer as the crosslinkable component in a Banbury mixer or the like at a temperature below the crosslinking temperature, or in a range of from 40° to 80°C, for example; forming the mixture to the desired shape using a roller mill or the like; then curing the shape by heating it to the crosslinking temperature, or in a range of from 100 to 140°C for example, for a sufficient time, or 5 to 15 hrs., for example.

The type of products which can be manufactured by the invention is not particularly limited, and include building materials such as tiles, wall coverings, floor coverings, and water-proof sheet materials. The present invention is particularly suitable for the manufacture of floor tiles and lengths of floor covering materials.

As described above, the self-crosslinking composition of this invention enables the crosslinking of elastomers containing diene or non-diene

groups and polymers containing many tertiary carbon atoms or alpha-carbon atoms using radicals generated from the decomposition of peroxide compounds which result from the oxidation of the oil in air while using no additional crosslinking agent or curing agent and using no special equipment for excluding air.

Other functions and effects of this invention are listed below.

1. In this invention, together with drying agent, the oil forms a film on the surface of the self-crosslinking composition. The generated radicals enhance the crosslinking of the polymer or elastomer without the need to exclude air or oxygen as is needed in the case of crosslinking using organic peroxides. That is, the crosslinking of polymer or elastomer can be conducted in air without using any additional crosslinking agent. ✓
2. The curing by hot air in an oven for crosslinking the self-crosslinking composition of this invention results in a stiff film having a non-contaminating property and a glossy surface. Although the film-forming reaction proceeds at a rather low speed compared with the crosslinking by peroxide, the reaction proceeds in presence of air even at a relatively low reaction temperature.
3. The curing in hot air described above avoids product deformation during the processing and forming stages, and results in a lower residual strain than that in rubber tiles formed by press-forming process or in polyvinyl chloride tiles formed by calender roll forming. As a result, such curing reduces the risk of shape change with time.

4. The solubility parameter (SP) of the oil used in this invention is preferably in a range of from 8.4 to 8.6. The SP value of elastomer and polymer as the crosslinking composition is also near the range of oil. Therefore, individual components can satisfactory dissolve each other. ✓

5. Some of the antioxidants and anti-ageing agents which may be contained in crosslinking polymer or elastomer inhibit the conventional crosslinking reaction by peroxide. However, the self-crosslinking compositions of this invention are not affected by such additives.

6. The products of this invention show particularly superior abrasion resistance, cigarette burn resistance, stain resistance, and give low charge voltage and high stiffness.

Accordingly, the present invention is suited to the manufacture of floor tiles and lengths of floor covering materials.

In the accompanying drawings:-

Fig. 1 is the infrared absorption spectrum of a composition before heating as prepared in Example 1,

Fig. 2 is the infrared absorption spectrum of the composition of Example 1 after heating to 100°C for 10 hrs,

Fig. 3 is the infrared absorption spectrum of a composition before heating as prepared in Example 2,

Fig. 4 is the infrared absorption spectrum of the composition of Example 2 after heating to 120°C for 10 hrs,

Fig. 5 is the infrared absorption spectrum of the composition of Example 3 after heating to 100°C for 2 hrs,

Fig. 6 is the infrared absorption spectrum of the composition of Example 3 after heating to 100°C for 10 hrs,

Fig. 7 is the infrared absorption spectrum of the composition of Example 4 after heating to 100°C for 2 hrs, and

Fig. 8 is the infrared absorption spectrum of the composition of Example 4 after heating to 100°C for 10 hrs.

Aspects of this invention are described below.

- (1) A self-crosslinking composition which comprises:
 - (a) an oil containing an unsaturated fatty acid and/or its ester,
 - (b) a drying agent, and
 - (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide.
- (2) A product obtained by shaping and crosslinking a self-crosslinking composition which comprises:
 - (a) an oil containing an unsaturated fatty acid and/or its ester,
 - (b) a drying agent,
 - (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and

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(d) a filler.

(3) A method for manufacturing a product by:
blending

- (a) an oil containing an unsaturated fatty acid and/or its ester,
- (b) a drying agent,
- (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
- (d) a filler,

to prepare a self-crosslinking composition;

forming the self-crosslinking composition into the desired shape at a temperature lower than the crosslinking temperature of the composition; and

crosslinking the shape at a temperature higher than the crosslinking temperature of the composition.

(4) A tile, for example a floor tile, obtained by forming and crosslinking a self-crosslinking composition which comprises:

- (a) an oil containing an unsaturated fatty acid and/or its ester
- (b) a drying agent,
- (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
- (d) a filler, (for example, calcium carbonate).

(5) A method for manufacturing a tile, for example a floor tile, by:
blending

- (a) an oil containing an unsaturated fatty acid and/or its ester,
- (b) a drying agent,

(c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
(d) a filler (for example, calcium carbonate),
to prepare a self-crosslinking composition;
forming the self-crosslinking composition into the required shape at a temperature lower than the crosslinking temperature of the composition;
and
crosslinking the shape at a temperature higher than the crosslinking temperature of the composition.

The present invention will now be described in more detail in the following Examples in which the symbol "%" designated "wt.%" unless otherwise specified.

Example 1

A 8.5 g of 1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.) and 1.5 g of boiled oil prepared from soya bean oil containing 0.01% of cobalt naphthenate were dissolved in 20 ml of industrial toluene. From the prepared solution, a film having a thickness ranging from 0.05 to 0.1mm was formed using the flow-and-spreading method. The film was then heated in an oven at 100°C for 10 hrs. Infrared absorption spectrometry and an extraction test were conducted on the film before and after the heating. The extraction test was conducted by placing the film and industrial toluene as the solvent into a Soxhlet extractor, processing the contents for 8 hrs, and then determining the insoluble matter. From a comparison of Figs 1 and 2, it was confirmed that the heating reduced the absorption band due to the

carbon-carbon double bond. The content of insoluble matter in the composition (film) after heating at 100°C for 10 hrs was 95%.

Example 2

A film was prepared as described in Example 1 but using 8.5 g of ethylene-vinyl acetate copolymer (containing 25% of vinyl acetate) (V505; supplied by Mitsubishi Petrochemical Co.) and 1.5 g of the boiled oil described in Example 1. The film was heated in an oven at 120°C for 10 hrs. Infrared absorption spectrometry was applied before and after the heating. From Fig. 3 and Fig. 4, it was confirmed that the heating reduced the absorption band due to the carbon-carbon double bond.

Example 3

Example 1 was repeated using 4.5 g of ethylene-vinyl acetate copolymer (containing 25% of vinyl acetate) (V505; supplied by Mitsubishi Petrochemical Co.), 4.5g of 1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.), and 1 g of the boiled oil described in Example 1. The film was heated in an oven at 100°C for 10 hrs. Infrared absorption spectrometry was applied to the film during and after the heating. From Fig. 5 and Fig. 6, it was confirmed that the heating reduced the absorption band due to the carbon-carbon double bond with time. The content of insoluble matter in the composition (film) after heating at 100°C for 10 hrs was 57%. (The extraction test was the same as in Example 1.)

Example 4

Example 1 was repeated using 4 g of ethylene-vinyl acetate copolymer (containing 25% of vinyl acetate) (V505; supplied by Mitsubishi Petrochemical Co.), 4 g of 1,2-syndioactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.), 1 g of the boiled oil described in Example 1, and 1 g of trimethylol propane tri-methacrylate (TMPT). From Figs 7 and 8, it was confirmed that the heating reduced the absorption band due to the carbon-carbon double bond with time. The content of insoluble matter in the composition after heating at 100°C for 10 hrs was 71%.

Example 5

A film was prepared by a similar procedure to Example 1 using 4 g of VLDPE (Tafmer P068; supplied by Mitsui Petrochemical Industries, Ltd), 4 g of 1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.), 1 g of the boiled oil described in Example 1, and 1 g of TMPT. The film was heated in a similar manner to that described in Example 1. Infrared absorption spectrometry and the extraction test were applied to the film.

TMPTMA

From the analytical results, it was confirmed that the heating reduced the absorption band due to the carbon-carbon double bond with time. The content of insoluble matter in the composition after heating was 55.4%.

When a composition comprising 8 g of VLDPE, 1 g of the boiled oil described in Example 1, and 1 g of TMPT was used, the content of insoluble matter became 14.3%. Consequently, the effect of the addition

of 1,2-syndiotactic polybutadiene having a crosslinking efficiency of 90 to 100% was confirmed.

From the above result, it was understood that the use of 1,2-syndiotactic polybutadiene having a high crosslinking efficiency provides a high curing speed (crosslinking speed) and that the use of an ethylene-vinyl acetate copolymer having a low crosslinking efficiency results in a small insoluble matter content and a slow curing speed. However, it was also confirmed that the use of 1,2-syndiotactic polybutadiene with acrylate and methacrylate which contain 1 to 4 functional groups accelerates the crosslinking reaction.

Example 6

Floor tiles were manufactured using the following composition:-

1,2-syndiotactic polybutadiene (RB801; supplied by Japan Synthetic Rubber Co.)	14%
Butadiene rubber (BR01; supplied by Japan Synthetic Rubber Co.)	2%
Soya bean oil (boiled oil containing 0.02% of cobalt naphthenate)	2%
Trimethylol propane methacrylate (TMPT)	2%
Calcium carbonate powder (under 150 mesh)	63%
Silicate (Wollastonite)	10%
Organic fiber reinforcing material (Santweb; supplied by Monsanto)	4%
Titanium white, other pigment	3%
Glycerin monostearate lubricant	0.5%

The above-listed components except the 1,2-syndiotactic polybutadiene and the butadiene rubber were preliminarily mixed in a Henschel mixer.

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After weighing such mixture, it was charged into a Banbury mixer along with the 1,2- syndiotactic polybutadiene and the butadiene rubber and blended at an initial temperature of from 50 to 60°C for 3 min. The resultant mixture was discharged (at a temperature of about 90 to 100°C). Then, the mixture was rolled by mixing rolls and embossing roll calender rolls to form a sheet of the required thickness. The sheet was cut to size and was put into an oven to cure at a temperature range of from 110 to 120°C for 10 to 12 hrs. After taking out the cured material, it was cut to a specific size to obtain the floor tiles.

Example 7

Floor tiles were manufactured in a similar manner to that described in Example 6 using the following composition:-

1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.)	12.0%
Ethylene-vinyl acetate copolymer (containing 25% of vinyl acetate) (V505; supplied by Mitsubishi Petrochemical Co.)	13.5%
Soya bean oil (boiled oil containing 0.02% of cobalt naphthenate)	3.0%
TMPT	1.5%
Fired clay (Translink#37; supplied by Engelhard Minerals)	10.0%
Water-containing silicate powder (Nipseal VN3; supplied by Nippon Silica Co.)	3.0%
Precipitated calcium carbonate treated by resin acid (Hakuenka O; supplied by Shiraisha Calcium Co.)	5.0%
Ground calcium carbonate powder (-150 mesh)	49.0%
Pigment	3.0%

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The floor tiles manufactured in this Example had the feel of rubber tiles, and had the following properties.

Specific gravity	1.71 g/cm ³	
Residual dent	6.1%	
Macburney dent	20°C	0.46mm
	45°C	0.69mm

Taber type abrasion tester (conforming to JIS K7204)

Test condition - H-18;	load of 1 kg; 1000 rpm
Abraded weight of 0.33 g;	abraded thickness of 0.07 mm
Cigarette proof	Favourable
Anti-stain property	Excellent
Charge voltage	150V (Floor polishing charge tester*, 20°C, 40% humidity)
Touch	Flexibility and elasticity resemble rubber tile.

data!

(*) Floor polishing charge tester is a charge tester which determines static charge potential generated during mechanical rubbing of the work (floor tile in this case) with a rubber roller. A conventional polyvinyl chloride composition called the "P-tile" was tested on the floor polishing charge tester under the condition described above, and the charge voltage was 1735V.

Example 8

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Cork powder, wood powder, and organic fiber were mixed into a binder at a blending temperature of 90 to 100°C without damaging the composition. For example, a floor tile having the following components was manufactured:-

1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.)	13.0%
Butadiene rubber (BR01; supplied by Japan Synthetic Rubber Co.)	2.0%
Soya bean oil (boiled oil containing 0.02% of cobalt naphthenate)	4.0%
TMPT	2.0%
Silicate (Wollastonite)	10.0%
Cork powder (FPF - 200 mesh)	7.0%
Ground calcium carbonate powder (-150 mesh)	62.0%

The above listed components except 1,2-syndiotactic polybutadiene and butadiene rubber were preliminarily mixed in a Henschel mixer. After weighing the mixture, it was charged to a Banbury mixer along with the 1,2-syndiotactic polybutadiene and the butadiene rubber and blended at a temperature range of from 90 to 100°C for 3 mins, following which then the resultant mixture was discharged. Then mixture was rolled by mixing rolls, and embossing roll calender rolls to form a sheet of the required thickness. The sheet was cut to size and was put into an oven to cure at a temperature range of from 110 to 120°C for 10 to 12 hrs. After taking out the cured material, it was cut to a specific size by a punching machine to obtain the floor tiles. The floor tiles had the properties given below. When the content of soya bean oil exceeded 20%, the obtained tile had an enhanced odor of fatty acid.

Specific gravity	1.60g/cm ³
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Residual dent 8.8%

Change in water-absorbing length (%)

+0.07% (X: along the calender flow direction)

+0.07% (Y: lateral to the calender flow direction)

Taber type abrasion tester (conforming to JIS K7204)

Test condition - H-18; load of 1kg; 1000rpm

Abraded weight of 1.28 g; abraded thickness of 0.31 mm

Static charge 200 - 300 V (Floor polishing charge tester, 20°C, 40% humidity)

Anti-stain property Excellent

Stiffness Determined by Taber type abrasion tester.

Result is summarized in Table 2.

Table 2 Result of stiffness measurement

Temperature	Stiffness	
	X: direction of calender flow	Y: lateral to calender flow direction
5	6695	6652
20	2612	2261
35	2022	1902

Example 9

An elongated length of floor covering material was manufactured using the following composition:-

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1,2-syndiotactic polybutadiene (RB810; supplied by Japan Synthetic Rubber Co.)	9.0%
Ethylene-vinyl acetate copolymer (containing 25% of vinyl acetate) (V505; supplied by Mitsubishi Petrochemical Co.)	16.5%
Boiled oil (soya bean oil containing 0.02% of cobalt naphthenate and 0.02% of zinc naphthenate)	3.0%
TMPT	1.5%
Water-containing silicate powder (Nipseal VN3; supplied by Nippon Silica Co.)	3.0%
Fired clay (Translink#37; supplied by Engelhard Minerals)	20.0%
Precipitated calcium carbonate treated by resin acid (Hakuenka O; supplied by Shiraishi Calcium Co.)	5.0%
Calcium carbonate heavy powder (-150 mesh)	29.0%
Cork particule (P-3, European normal)	10.0%
Pigment	3.0%

The above-listed components were mixed in a Banbury mixer to form square pellets having a size of 3 x 3mm. The pellets were heated by hot air to 90 to 100°C, then they were supplied to a calender roll machine to press-weld them to a Tetron nonwoven fabric or a victoria lawn which was treated by SBR latex. The treated fabric was hung down from louvers passing through an oven to cure at 120 to 130°C for 12 to 14 hrs.

Thus obtained elongated floor covering material had the following properties.

Specific gravity	1.31g/cm ³
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Residual dent	15%
Change in water-absorbing length	(%)
+0.13% (X: along the calender flow direction)	
+0.11% (Y: lateral to the calender flow direction)	
Taber type abrasion tester (conforming to JIS K7204)	
Test condition - H-18; load of 1 kg; 1000 rpm	
Abraded weight of 1.50 g	
Cigarette proof property	Favourable to similar degree with ordinary linoleum
Anti-stain property	Excellent
Static charge	98V (Floor polishing charge tester, 27°C, 21% humidity)

CLAIMS

1. A self-crosslinking composition comprising:
 - (a) an oil containing an unsaturated fatty acid and/or its ester,
 - (b) a drying agent, and
 - (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide.

2. A product obtained by shaping and crosslinking a self-crosslinking composition which comprises:
 - (a) an oil containing an unsaturated fatty acid and/or its ester,
 - (b) a drying agent,
 - (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
 - (d) a filler.

3. A product as claimed in claim 2, in the form of floor covering material or wall covering material.

4. A method for manufacturing a product, comprising:
blending
 - (a) an oil containing an unsaturated fatty acid and/or its ester,
 - (b) a drying agent,
 - (c) a polymer and/or an elastomer capable of being crosslinked by a peroxide, and
 - (d) a filler,to prepare a self-crosslinking composition;

forming the self-crosslinking composition into the desired shape at a temperature lower than the crosslinking temperature of the composition; and crosslinking the shape at a temperature higher than the crosslinking temperature of the composition.

5. A method as claimed in claim 4, wherein the product is a floor covering material or a wall covering material.
6. A composition as claimed in claim 1, substantially as hereinbefore described in any of the Examples.
7. A product as claimed in claim 2, substantially as hereinbefore described in any one of the Examples.
8. A method as claimed in claim 4, substantially as hereinbefore described in any one of the Examples.

Search Examiner
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Relevant Technical Fields

- (i) UK Cl (Ed.N) C3M (MFC, MXC)
(ii) Int Cl (Ed.6) C08K; C08L

Date of completion of Search
9 AUGUST 1995

Databases (see below)

- (i) UK Patent Office collections of GB, EP, WO and US patent specifications.

Documents considered relevant following a search in respect of Claims :-
1-8

- (ii) ONLINE: WPI

Categories of documents

- X: Document indicating lack of novelty or of inventive step. P: Document published on or after the declared priority date but before the filing date of the present application.
Y: Document indicating lack of inventive step if combined with one or more other documents of the same category. E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.
A: Document indicating technological background and/or state of the art. &: Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages	Relevant to claim(s)
X	GB 2205104 A (K E VALLALAT) Claim 1	at least Claims 1, 2, 3
X	GB 1518324 (UNION CARBIDE) eg. Table VI on page 15; page 2, lines 17-18	at least Claims 1, 2, 3
X	GB 0711470 (UK CHEMICALS) Table on page 4 and examples	at least Claims 1, 2, 3
<i>requested</i> → X	US 4746695 (BERLY) Examples	at least Claims 1, 2, 3
X	WPI Abstract Accession No 89-124675/17 & HU T47622 A (BUDALAKK etc) 28 March 1989 (see abstract)	at least Claim 1
X	WPI Abstract Accession No 83-770227/38 & SU 975761 A (MOSC CHEM PAINT) 23 November 1982 (see abstract)	at least Claim 1

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).